

**Trace Metals by Graphite Furnace Atomic Absorption**  
**EPA 200.9 Revision 2.2**

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Facility Name: \_\_\_\_\_ VELAP ID \_\_\_\_\_

Assessor Name: \_\_\_\_\_ Analyst Name: \_\_\_\_\_ Inspection Date \_\_\_\_\_

**Relevant Aspect of Standards****Method  
Reference****Y****N****N/A****Comments**

Records Examined: SOP Number/ Revision/ Date \_\_\_\_\_ Analyst: \_\_\_\_\_

Sample ID: \_\_\_\_\_ Date of Sample Preparation: \_\_\_\_\_ Date of Analysis: \_\_\_\_\_

For dissolved elements, are samples filtered using 0.45  $\mu\text{m}$  pore diameter membrane filters and then preserved using (1+1) nitric acid to pH <2?

8.2

For total recoverable elements, are samples NOT filtered and acidified using (1+1) nitric acid to pH&lt;2?

8.3

For total recoverable elements, are samples held for 16 hours after preservation and then verified to be pH<2 just prior to withdrawing an aliquot for processing? (If pH is not <2, additional acid is added and sample is held for an additional 16 hours and rechecked.) *ONLY ENFORCED FOR DRINKING WATER per CFR.*8.3,  
40 CFR  
141.23 k.1

Are solid samples stored at 4°C?

8.4

For silver, are wastewater samples diluted prior to digestion if silver is &gt;0.1 mg/L?

1.6

Is high-purity grade argon (99.99%) used during the atomization of selenium, sheathing the furnace tube when in operation, and during furnace cleanout?

6.1.3

Is an alternate gas mixture containing hydrogen 5%-argon 95% used as a continuous gas flow environment during the dry and char furnace cycles?

6.1.4

Is the hot plate or block digester capable of maintaining a temperature of 95°C?

6.3, 6.4

Is labware cleaned using 20% nitric acid or a mixture of dilute  $\text{HNO}_3$  and  $\text{HCl}$ , followed by rinsing with DeI water?

6.10

Are all acids ultra-high purity grade or equivalent?

7.1

Is reagent water ASTM Type 1? Conductivity at 25°C  $\leq$  0.0555  $\mu\text{S}/\text{cm}$ ; Resistivity at 25°C  $\geq$  18; TOC  $\leq$  50  $\mu\text{g}/\text{L}$ ; Sodium  $\leq$  1  $\mu\text{g}/\text{L}$ ; Chloride  $\leq$  1  $\mu\text{g}/\text{L}$ ; Total silica  $\leq$  3  $\mu\text{g}/\text{L}$ 7.4 (See  
ASTM  
D1193-06)

Notes/Comments:

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Relevant Aspect of Standards	Method Reference	Y	N	N/A	Comments
Is the linear dynamic range established for each analyte using at least six different concentration standards?	9.2.2				
Is the MDL established using concentrations 2 to 3 times the estimated instrument detection limit, and by analyzing seven replicates through the entire procedure?	9.2.4				
For dissolved analytes or direct analysis of total recoverable analytes in drinking water, are samples prepared by adding 0.4 mL (1+1) HNO <sub>3</sub> to 20 mL of sample?	11.1.1				
For total recoverable analytes (aqueous samples other than drinking water with <1 turbidity), are samples prepared by digesting 100 mL sample with 2 mL (1+1) HNO <sub>3</sub> and 1 mL (1+1) HCl at 85°C? (Smaller volumes may be used as long as the proportions are correct.) Samples are reduced to 20 mL and then covered and refluxed for 30 minutes.	11.2.3				
After digestion of aqueous samples, are samples allowed to cool and returned to a volume of 50 mL with reagent water?	11.2.6				
For solids, is percent solids determined by weighing a portion of at least 20 g (50-100 g if percent moisture is >35%), recording wet weight, drying at 60°C, and recording dry weight? % solids= (DW/WW) × 100	11.3.1				
For solids, is an aliquot of 1 ± 0.01g of dried sample extracted with 4 mL of (1+1) HNO <sub>3</sub> and 10 mL of (1+4) HCl at 95°C? The sample is heated and refluxed 30 min.	11.3.3, 11.3.4				
For solids, are extracts allowed to cool and brought up to 100 mL with reagent water?	11.3.5				
Are the instrument and hollow cathode lamp allowed to warm up for at least 15 minutes? (If using an electrodeless discharge lamp, allow 30 minutes.)	10.3				
Does the calibration include a calibration blank and at least three calibration standards?	11.4.4				
Notes/Comments:					

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Relevant Aspect of Standards	Method Reference	Y	N	N/A	Comments
Are calibration standards and the calibration blank prepared using the appropriate acid diluent? For dissolved elements or direct analysis of drinking water with turbidity <1 NTU, use 1% HNO <sub>3</sub> . For total recoverable in water, use 2% HNO <sub>3</sub> and 1% HCl. For total recoverable in solids, use 2% HNO <sub>3</sub> and 2% HCl.	7.9, 7.10.1				
Is the laboratory reagent blank carried through the entire sample preparation with each batch of 20 or fewer samples?	7.10.2, 9.3.1				
Does the laboratory reagent blank contain less than 10% of the analyte level observed in samples and less than 2.2 times the analyte MDL?	9.3.1				
Is the laboratory fortified blank prepared to provide a final concentration which will produce an absorbance of approximately 0.1 for each analyte, and is it carried through the entire sample preparation?	7.10.3				
Is the laboratory fortified blank within 85-115% recovery?	9.3.3				
Is the rinse blank prepared in a ratio of 1 mL conc. HNO <sub>3</sub> and 1 mL conc. HCl to 1L water?	7.10.4				
Is the instrument performance check solution prepared from the same stock as the calibration standards?	7.11				
Are the instrument performance check solution and a calibration blank analyzed immediately after calibration, after every 10 <sup>th</sup> sample, and at the end of the run?	9.3.4				
Is the instrument performance check solution immediately following calibration within 5% of calibration, and are subsequent analyses within 10% of calibration?	9.3.4				
Is the quality control sample obtained from an outside source AND prepared in the same acid mixture as the calibration standards?	7.12				
Is the recovery of the quality control sample within 10%?	9.2.3				
Is a spike analyzed for at least 10% of samples, with an acceptable range of 70-130%?	9.4.2, 9.4.3				
Notes/Comments:					

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Relevant Aspect of Standards	Method Reference	Y	N	N/A	Comments
For dissolved analytes, are results reported directly from the instrument with allowances for dilutions?	12.2				
For total recoverable aqueous analytes, are results multiplied by the dilution factor 0.5 if using a 100 mL aliquot to produce the 50 mL final solution?	12.3				
For total recoverable analytes in solids, are results calculated using the following formula? Sample concentration, dry weight basis (mg/kg) = $(C \times V \times D) / W$ where C= extract concentration (mg/L), V= extract volume (L), D= dilution factor, and W= weight of sample aliquot extracted	12.4				

Notes/Comments: